THE DETERMINATION OF SULPHATES IN WATER.

The Use of Benzidine Hydrochloride.

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THE majority of the waters of Kansas often contain considerable quantities of calcium sulphate, whose exact and quick determination is very often necessary. Especially is this true in the examination of boiler waters. The usual procedure for this determination is to precipitate the calcium sulphate as barium sulphate, filter, and weigh. This operation requires from four to twelve hours to complete, depending upon the quantity of calcium sulphate in the water. Where the content in calcium sulphate is desired to be known on short notice, therefore, this method will not meet the demand. The benzidine method, however, will, as the results herein contained show.

The method consists in precipitating the sulphates by means of benzidine hydrochloride—$C_{12}H_8(NH_2)_2 \cdot 2HCl$—as benzidine sulphate, which being insoluble in water is filtered off, suspended in water, and titrated in the hot with standard alkali, using phenolphthalein as indicator. This titration is possible by the very weak basic properties of benzidine. The whole operation can be completed in from fifteen to thirty minutes.

The method is not original with me, having been proposed by Raschig (Zeit. angew. Chem. 1903, 16:617) and investigated by Friedam and Nydegger (Zeit. angew. Chem. 1907, 19:9), whose suggestions I have followed and adapted and modified to suit water-analysis conditions.

After a few trial experiments the following method was decided upon as the best procedure for water analysis: To 250 cc. of water (less if the SO_4 content is greater than 500 parts per million) add 10 cc. of a 1 per cent solution of hydroxylamine hydrochloride (more if the iron content of the water is very high) and 20 cc. of benzidine hydrochloride. Stir vigorously and allow the silky white precipitate to settle. Filter on a disc of black ribbon filter paper in a Gooch crucible, *with suction.* Wash with cold distilled water twice (about 25 cc.), and drain precipitate thoroughly. Transfer the precipitate to the original beaker, add water, and heat to boiling. Titrate with N/20 NaOH, using phenolphthalein as the indicator. Parts per million $SO_4 = 9.6 \times$ cc. N/20 NaOH.

The benzidine hydrochloride solution is made up as follows: Place 8 grams of benzidine in an agate mortar, and add enough water to make a paste. Wash the paste into a one-liter flask, add 10 cc. concentrated HCl, and make up to the mark. Filter if necessary. 1 cc. = .0026 grams SO_4.

This method and the barium chloride method usually employed were used in the determination of sulphates on several hundred waters. The results by the two methods agreed very closely, as the data in the following table shows:

Comparison of results obtained with Barium Chloride and Benzidine.

Results expressed in parts per million.

Lab. No.	SO_4 by $BaCl_2$.	SO_4 by benzidine.	Difference.
6678	80.6	84.5	+3.9
6682	76.5	80.6	+4.1
6723	254.2	257.3	+3.1
6729	87.6	96.0	+8.4
6742	68.7	76.8	+8.1
6804	45.4	40.3	—5.1
6887	584.7	583.7	—1.0
6942	62.2	63.4	+1.2
7081	29.6	32.6	+3.0
7082	173.2	180.5	+7.3
7084	343.9	341.5	—2.4
7087	101.2	107.5	+6.3
7114	51.0	46.0	—5.0
7128	19.8	15.4	—4.4
7139	38.8	38.4	—0.4
7142	24.7	19.2	—5.5
7144	48.0	46.0	—2.0
7148	16.2	21.0	+4.8
7155	26.3	25.0	—1.3
7156	51.8	63.4	+1.6

Several precautions are necessary in order to obviate difficulty in carrying out the method. The precipitate should not be allowed to stand too long, for the silky precipitate turns to flakes on standing, which only dissolve after considerable boiling. The suction should be strong enough to remove the last traces of the mother liquid before any wash water is added. If it is not, too high results will be obtained.

After transferring the precipitate the water should be heated, preferably until all the precipitation is dissolved. Although this is not always necessary, it is sometimes a safe procedure to insure easier splitting off of the HCl groups.